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Anti-wear and Friction-reducing Characteristics of Two Kinds of Ceramic Additives

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Abstract. Two kinds of ceramic additives have been developed that one is the serpentine particles and another is a blend of serpentine particles and catalyst. The tribological properties of the addition of different additives are investigated through a series of friction and wear experiments. Wear surface and the composition of the tribofilm were examined by SEM, EDS and XPS. In case of single serpentine additive, tribo-film can be formed gradually on the worn metal surface. The friction coefficient is about 0.11. The tribofilm mainly consists of Mg and Si elements transferred from the additive. This can compensates part of wear mass loss, avoids the direct contact of the two rubbing surfaces, and thus effectively improves the anti-wear characteristics. In case of the blend oil additive, the tribo-film formed obviously on the worn surface in the initial stage and no obvious film at end of the test. However, the friction coefficient can lower even to 0.007~0.008 compared with the above experiment. The worn surface becomes very smooth. Chemical analysis shows that there is a very thin film of carbon concentration with thickness of 30~50nm on the worn surface. Existence of the very thin carbon-concentrated film and mirror-like surface generates super low friction coefficient.

Introduction

Friction and wear is a common phenomenon for mechanical equipments in operation. It is detrimental, leading to unwanted clearances between the moving components, additional freedom of movement, increased mechanical loading, more rapid wear, and ultimately system failure. At present, there are four kinds of methods to reduce friction and wear: (a) Surface chemical treatment, such as nitriding, carburizing, etc; (b) Deposition of a wear-resistant protective film on the surface of metal parts, such as TiN, diamond-like coating (DLC)[1] etc; (c) Addition of the anti-wear additives to lubricant to reduce wear in the process of friction and wear, etc[2-4]. One of the most interesting phenomena is that very fine wear particles are often produced in wear of ceramics and they are trapped and agglomerate to form a new surface film within the contact area[5-7]. They have been called the tribofilm, transfer layer. It has been also reported that such agglomerated wear particles control friction and wear.

By means of it, some special selected kinds of ceramic particles as oil additives are added into the lubricating oil. During the friction and wear, the additive can transfer continuously into the rubbing contact surface to recondition the worn surface. Therefore, the behavior of the added particles is recognized to be very important to understand the wear mechanism and design high wear resistant material or optimum operating conditions. In the paper, two kinds of ceramic additives have been developed. The influence of the addition of different additives in the super-fine particle on various physico-chemical properties of $45^{\#}$ steel friction pairs are investigated through a series of friction and wear experiments.

Experimental Details

The additive of serpentine and catalyst were silicates. The formula of serpentine is $Mg_3Si_2O_5(OH)_4$. The additive of serpentine and catalyst was fabricated respectively. The additive was first pulverized into particle with a size of less than 0.5mm, then uninterruptedly grinding by planetary ball-milling machine for 48h into a fine particle with the size smaller than 5 μm. Afterwards, the

additive particles were demoistured for 3h heat treatment at 120ºC. Silane coupling agents A1120 and titanate coupling agents NXT-105 were used as surface dispersants. The additive and dispersants were put into in acetone. The amount of surface modifier is 5% (wt) of the additive. The attained mixed solutions were ultrasonic oscillated for 20 min. Then the surface modified serpentine particles were obtained after the evaporation of acetone. Finally, the surface modified additive of serpentine (2%wt) is added into the lubricant oil. The blend of serpentine particles and catalyst (total 2%wt) with mass ratio of 70:30 is added into the lubricant oil.

Fig.1 schematically shows a wear test method employed in the present study, in which a stationary, loaded rub against a rotating disc. The rub was 10×10×16mm, and the disc was 10mm thick and 42mm in diameter. The material used for both the rub and disc was quenched and tempered 0.45 mass% carbon steel having a hardness of HRC43~45. The surface roughness of the specimens was Ra 1.0μm. The specimens were cleaned ultrasonically in acetone prior to testing.

The experiment was interrupted during the test to observe surface topography. The worn surface topography was examined by Philips XL-30 scanning electronic microscope (SEM). The composition on the wear surface was analyzed by energy dispersive X-ray analysis spectrometer (EDAX). XPS measurements were made on the worn surface of only the discs to determine the elemental composition on the worn surface and just beneath (etching surface for at most 24min).

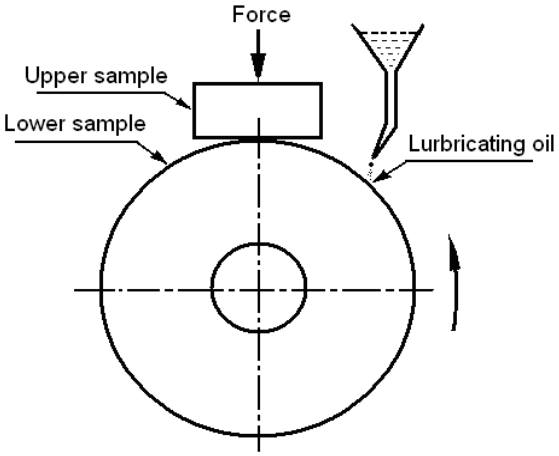

Fig. 1 Schematic diagram of wear test

Results

Serpentine Additive. The coverage of the steel worn surface after 10h testing is obvious as shown in Fig.2 (a). The steel worn surface becomes smoother. This suggests that the asperity peaks on the steel surface are removed. With increasing time, more and more additive is transferred onto the worn surface and it covers a large stretch of the grooves and the crevices as shown in Fig.2 (b). The very fine additive particles are trapped and agglomerated to form a new surface film within the contact area. The additive deforms, stretches and flows along the sliding direction on the steel surface, and the protecting coating is non-uniform and discontinuous. This means that the additives can be deformed and pulverized under the high shearing stress in the contacting zone. Most part of the surface is covered by tribofilm after 30h, even though a very small patches of metal surface is not covered in the local micro-area as shown in Fig.2(c). The worn surface is relatively smooth and fully covered by tribofilm at end of the test of 100h as shown in Fig.2 (d).

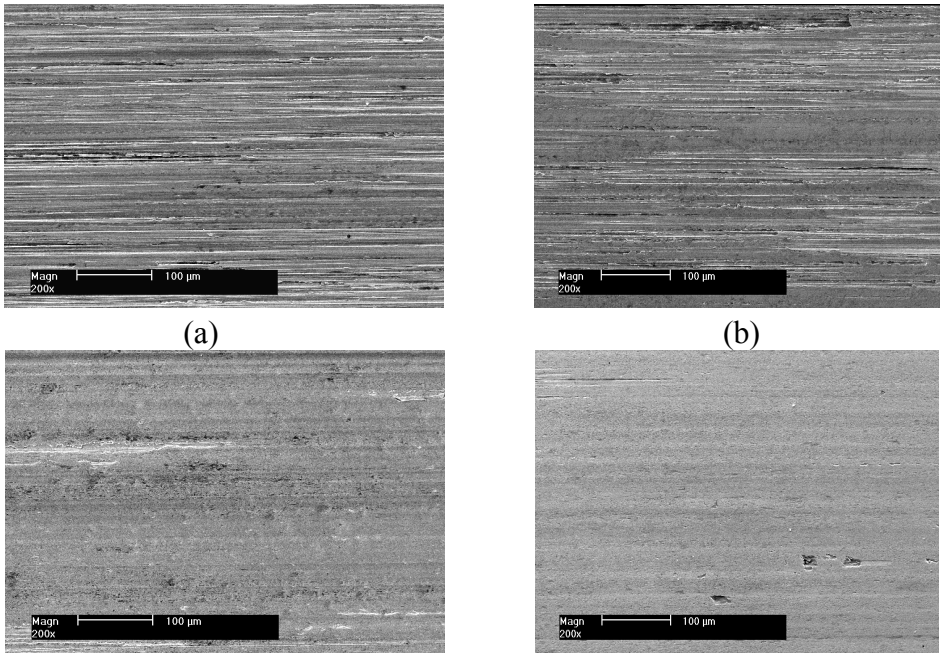

Fig. 2 SEM micrographs of a protecting coating after (a) 10h, (b)20h, (c) 30h, (d) 100h

A higher magnification SEM image of a pit in the tribofilm shows it to be thick and structured (Fig.3). Substantial tribofilm formation is observed both in the SEM image (Fig.3) and the EDX spectra showing magnesium and silicon in the wear surface (Fig.4).

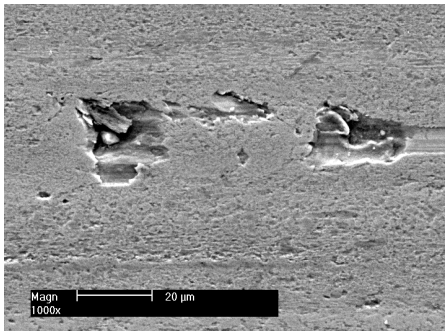

Fig. 3 SEM image of surface film

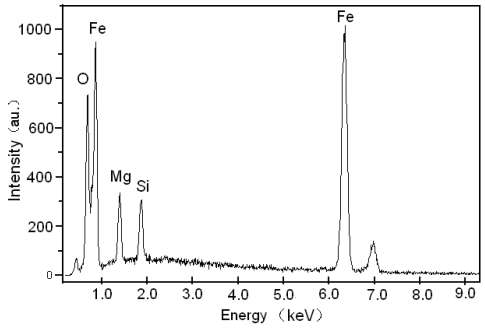

Fig. 4 EDX spectrum of surface film

The analysis of post-test samples was carried out to quantify the elements present on the worn surface and to define the species present on the near surface of the tribofilm listed in table 1. XPS result shows that the tribo-film mainly consists of Mg, Si, Fe, C, O elements. For middle steel (0.45% carbon), the Si content is about 0.2%-0.3%, and there is no magnesium element. So, we can deduce that magnesium and silicon mainly transferred from the serpentine additive [$Mg_3Si_2O_5(OH)_4$] during friction and wear.

Tab. 1 XPS quantification in at% of tribofilm formed on the sample surface（100h）

Etching time /min	Mg	Si	Fe	C	O
0	0.89	5.89	3.24	55.02	34.94
12	13.78	9.41	16.21	15.07	45.52
24	15.38	9.32	17.86	11.50	45.94

Mixed oil additives. Fig.5 (a) shows SEM images of the initial surface and Fig. 5(b) shows the SEM images of wear surface after 20h. It can be seen that surface smoothing proceeds by wear of asperity peaks of metals. A higher magnification SEM image of a thin film in the metal surface is

shown in Fig.5(c). The worn surface is covered by a thin tribofilm on the peaks and valleys of the surface profile of the initial surface. There still remain the surface scratches of the initial surface. However, an even surface is shown in Fig.5 (d) after 60h. Here, we shall concentrate on the position of the smooth surface formed after wear. Furthermore, in order to clarify the microstructure of the smooth surface of metals in sliding contact, the local signification image is shown in Fig.5 (e). The wear surface is fully covered by a thin tribo-film between the peaks and valleys of the initial surface.

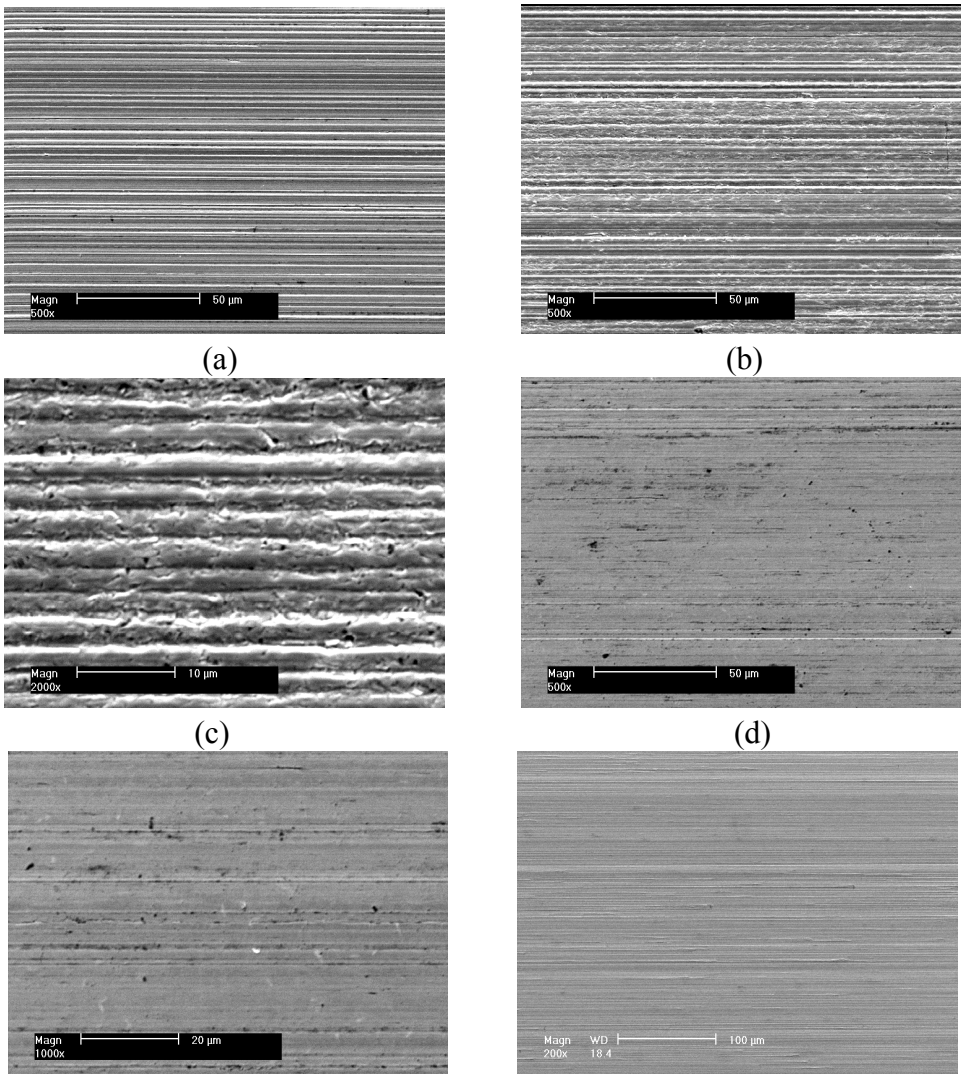

Fig. 5 SEM micrographs of worn surface after(a) 0h, (b)20h, (c) high signification of (b), (d) 60h, (e) high signification of (d) , (f) 100h

Table 2 XPS quantification in at% of tribofilm formed on the sample surface (100h)

Etching time /min	Si	Fe	C	O
0	2.92	4.23	63.77	28.67
6	0.28	38.56	23.46	37.34
12	0.25	44.41	18.03	36.73

Almost the whole wear surface becomes very smooth at 100h shown in Fig.5 (f). There is only some shallow plough and some hallows on the wear surface. The surface roughness is about 0.09μm as shown in Fig.6.

The analysis of post-test samples was carried out to quantify the elements present on the worn surface and to define the species present on the near surface of the tribofilm listed in table 2. It can be seen that the main composition elements of the worn surface were Fe, C, O, Mg and Si. There was no Mg element on the worn surface after 100h and Si content is very low. However, the content of C and O elements are very high. Tribofilm deposited on the metal surface did not form thick enough films to prevent the surfaces from direct contact. But existence of the rich-carbon and relatively mirror-like layer is the main factor of lower friction coefficient.

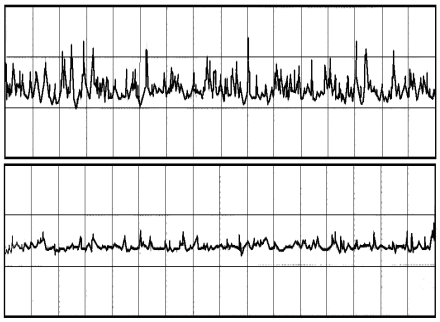

Fig. 6 Surface profiles of initial surface (upper) and smooth wear surface (lower)

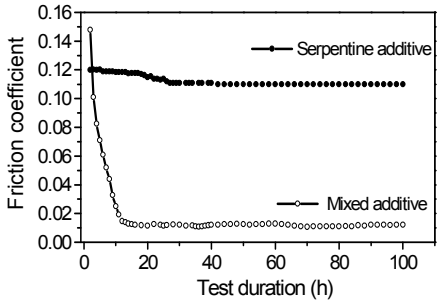

Fig. 7 Variation of friction coefficient with test duration

Discussion

Friction characteristics with serpentine additive and blend of additive are shown in Fig.7. In case of serpentine additive, as can be seen, the additive gradually transferred onto the worn surface in the first 30h, shown in Fig.2 (a) and Fig.2 (b). The friction coefficient drops slowly. In fact, the additive can transfer onto some area of the metal surface in the initial stage of the test, shown in Fig.8. At the beginning of the test run, the oil film is not stable and the tribofilm maybe only forms on the local surface. Metal-to-metal contact takes place and the friction coefficient is about 0.12. With increase of time, the additive can gradually transfer onto the worn metal surface, give rise to a slow decrease in friction coefficient. This also can compensate wear mass loss in part. When both of the upper and lower worn surface was fully covered by a thin, smooth tribofilm, shown in Fig.2(c)~(d) and Fig.9. Tribofilm-to-tribofilm contact takes place instead of metal-to-metal contact. The friction coefficient keeps stable about 0.105 to the end of the test.

In case of the mixed additive, the additive gradually transferred onto the worn surface in the first 20h, shown in Fig.5 (a)~(b). However, the friction coefficient drops sharply from 0.15 to 0.02 in the first 10h, and decreases slowly to 0.01 at 20h compared with that of the single serpentine additive. Friction coefficient also reaches its stable value in a much shorter time. Friction coefficient fluctuate very slowly and even to 0.007-0.008 during friction and wear test. The worn surface was mirror-like with fairly low surface roughness, shown in Fig.5 (d)~(f). Friction coefficient maintains stable about 0.01 to the end of the test.

One of the most important aspects of oil additive is its morphology change during wear and friction. After 100h test duration, particles were extracted from the used lubricating oil. For single additive, there is little morphology change before and after test, shown in Fig.10. However, the morphology of the wear debris turns into ball from irregular particles for mix additive shown in Fig.11. There is no obvious deformation for the spherical wear debris during wear and friction test. This suggests that the spherical wear debris possesses enough hardness and strength to sustain the friction load. In the same time, the spherical wear debris acted as a plenty of rolling balls between the two contacting surfaces. That turns the sliding friction condition into rolling friction condition. Once the enough spherical wear debris generates between the friction pairs, friction coefficient drops greatly and can maintain a very lower level during the wear and friction test. This illustrates that thin tribo-films caused by different additives possess great different characteristics. The tribofilm caused by serpentine additive aviods direct contact of the mating surface. So it has good anti-wear property. While the tribofilm caused by blend additive reduces the contact resistance of the contact interface.

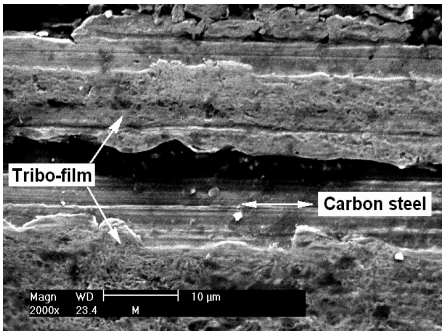

Fig. 8 SEM micrographs of worn surface(5h)

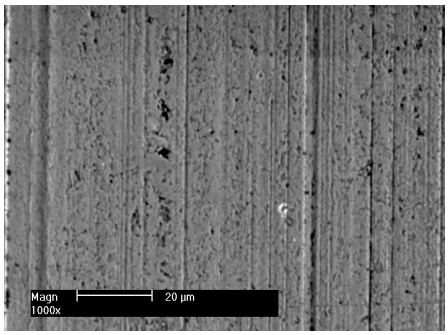

Fig. 9 The worn surface of the upper sample(100)

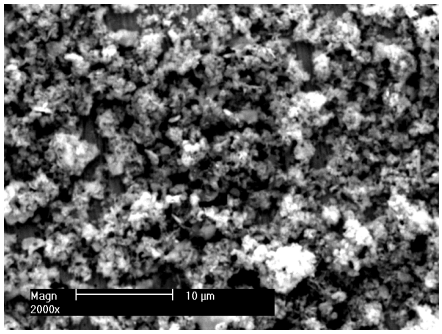

Fig.10 Wear debris of single additive (100h)

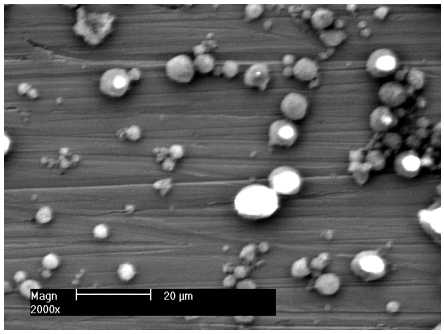

Fig.11 Wear debris of the mixed additives (100h)

Summary

(1). The serpentine additive can continuously transfer onto the wear surface and gradually form a thick tribofilm. Friction coefficient with the serpentine additive is about 0.11. The tribofilm on the wear surface compensates part of wear mass loss, avoids the direct contact of the two rubbing surfaces, and thus effectively improves the antiwear performance.

(2). The blend additive can transfer onto the wear surface in the initial stage and form a very thin tribofilm in the end of test compared with the single serpentine additive. Friction coefficient with the blend additive is fairly lower or even to 0.007-0.008. Existence of the very thin carbon-concentrated film and the mirror-like wear surface generates super low friction coefficient. Therefore, the blend additive possesses excellent friction-reducing characteristics during friction and wear test.

Acknowledgements

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